
विस्फोटक और आतिशबाज़ी संयोजन के लिए
क्यूप्रिक ऑक्साइड — विशिष्ट

Cupric Oxide for Explosives and
Pyrotechnic Composition —
Specification

ICS 71.060.20; 71.100.30

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Cupric Oxide, Cu(II)O , is an inorganic chemical which is available commercially in the form of black powder. It is used in varied applications like animal feed supplements, anti-fouling paints, bactericides and fungicides, blue glass and blue jewel manufacturing, catalyst in industries, coloring agent in ceramic and glass industries, desulphurizing agent for oils, fireworks/pyrotechnic compositions, friction materials for brakes and clutches, galvanic electrodes, making ceramics and fibers, medicines, metal adhesive curing agent, and mordant in dyeing and printing textile fabrics etc.

The technical or reagent grade of cupric oxide is used for the purpose of fireworks and pyrotechnics. Cupric oxide forms an explosive mixture with powdered aluminum; anilinium perchlorate; magnesium; and phthalic anhydride, which explodes when heated.

There is no ISO standard on the subject.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CUPRIC OXIDE FOR EXPLOSIVES AND PYROTECHNIC COMPOSITION — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of sampling and methods of test for technical and reagent grade cupric oxide for use in explosive and pyrotechnic composition.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publications, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
4905 : 2015	Random sampling and randomization procedures (<i>first revision</i>)
10081 : 1981	Terms relating to commercial explosives, pyrotechnics and blasting practices

3 TERMINOLOGY

For the purpose of this standard, the terms and definitions given in IS 10081 shall apply.

4 GRADES

The material shall be of the following grades:

- a) Grade 1 — Analytical reagent grade.
- b) Grade 2 — Technical grade.

5 REQUIREMENTS

5.1 Form and Condition

The material shall be in the form of a dry powder and shall be free from adulterants and foreign matter. It shall be dark brown / brownish black / black amorphous powder. Cupric oxide is a monoclinic crystal, its mineral form is also known as tenorite.

5.2 The material shall comply with requirements given in Table 1.

Table 1 Requirements for Cupric Oxide for Explosives and Pyrotechnics Industry
(Clause 5.2)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Clause
		Technical Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Sieving :			A-1
	a) Retained on 150 µm IS sieve, percent, <i>Max</i>	2	Not applicable	
	b) Retained on 45 µm IS sieve, percent, <i>Max</i>	Not applicable	0.5	
ii)	Volatile matter, percent, <i>Max</i>	0.2	0.2	A-2
iii)	Total copper, percent, <i>Min</i>	78.0	79.0	A-3
iv)	Cuprous oxide, percent, <i>Max</i>	0.50	0.05	A-4
v)	Cupric oxide, percent, <i>Min</i>	98.0	99.0	A-5
vi)	Chloride as Cl, percent, <i>Max</i>	0.10	0.01	A-6
vii)	Sulphate as SO ₄ , percent, <i>Max</i>	0.10	0.01	
viii)	Iron as Fe, percent, <i>Max</i>	0.05	0.01	A-7
ix)	Lead as Pb, percent, <i>Max</i>	0.02	0.01	
NOTE — Requirements may also be agreed to between purchaser and supplier.				

6 PACKING

25 kg or 40 kg of material shall be first packed in polyethylene bags of not less than 0.087 5 mm (350 gauge) thickness, then encased in MS drum and closed tightly.

Packaging may also be done according to the agreement between the purchaser and the supplier.

7 MARKING

7.1 The following information shall be marked legibly and indelibly on each container:

- a) Name of the material;
- b) Grade;
- c) Name of the manufacturer;
- d) Batch number; and
- e) Net mass of contents.

7.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

8 STORAGE

The material shall be stored in a cool and dry place to prevent dampness. The material shall be kept isolated from strong acids or food. In case of fire, water shall be used to extinguish it.

9 SAMPLING

The method of drawing the representative samples of the material and criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(Table 1)

METHODS OF TEST FOR CUPRIC OXIDE FOR EXPLOSIVE AND PYROTECHNICS INDUSTRY

A-1 DETERMINATION OF RETENTION ON SIEVE**A-1.1 Apparatus**

A-1.1.1 Test Sieves, of aperture size 150 μm and 45 μm IS Sieve (7.6 cm dia $\times$ 3.8 cm deep brass / stainless steel frame fitted with SS wire woven cloth).

A-1.1.2 Camel Brush (No.8).

A-1.1.3 Container, of suitable size to accommodate the test sieve. It should be half-filled with acetone.

A-1.1.4 Analytical Balance, 0.1 mg accuracy.

A-1.1.5 Air Oven, maintained at 105°C

A-1.2 Procedure

Transfer 5 g of accurately weighed sample to the test sieve. Keep the test sieve with test portion under a flow of stream of tap water with brushing to assist the powder to elute through the sieve opening until no more particles pass through the test sieve. Drain the water from the test sieve and oscillate the test sieve downwards and upwards in the acetone container so that the screen surface passes just under and above the level of acetone. Drain the acetone from the test sieve. Place the test sieve with the residue in an air oven at 105°C for about 30 min. Cool it, collect and weigh the residue.

A-1.3 Calculation

Calculate the residue (retention) on sieve, as percent by mass, using the following formula:

$$\text{Retention of sieve, percent by mass} = \frac{M_1}{M_0} \times 100$$

where

M_1 = Mass of residue on the test sieve, in g, and

M_0 = Mass of the sample taken for test, in g.

A-2 DETERMINATION OF VOLATILE MATTER**A-2.1 Apparatus**

A-2.1.1 Analytical Balance, 0.1 mg accuracy.

A-2.1.2 Air Oven, maintained at 105 $\pm$ 2°C.

A-2.1.3 Weighing Bottle, squat form, wide mouthed with ground glass stopper having 60 mm diameter and 40 mm height.

A-2.1.4 Desiccator, containing silica gel as desiccant.

A-2.2 Procedure

Heat the clean weighing bottle with the stopper removed in an air oven maintained at 105 $\pm$ 2°C for 1 h. Allow to cool in a desiccator. Insert the stopper and weigh it accurately nearest to 0.1 mg (W_1). Take 5 g of the sample in the weighing bottle, insert the stopper and weigh to the nearest 0.1 mg (W_2). Heat it, with the stopper removed, in the oven at 105 $\pm$ 2°C for 2 h. Allow to cool in a desiccator. Insert the stopper and weigh to the nearest 0.1 mg (W_3).

A-2.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(W_2 - W_3)}{(W_2 - W_1)} \times 100$$

where

W_1 = weight of weighing bottle + stopper,

W_2 = weight of weighing bottle + stopper with the sample before drying, and

W_3 = weight of weighing bottle + stopper with sample after drying.

A-3 DETERMINATION OF TOTAL COPPER**A-3.1 Principle**

Copper is determined by titration of the liberated iodine after addition of potassium iodide to a weakly acidic solution. Difficulties with absorption of iodine on the cuprous / cupric iodide precipitate are avoided by the addition of ammonium thiocyanate.

A-3.2 Apparatus

A-3.2.1 Analytical Balance, 0.1 mg accuracy.

A-3.2.2 Volumetric Flask, 100 ml and 1 000 ml.

A-3.2.3 Conical Flask, 250 ml.

A-3.2.4 Beaker, 250 ml.

A-3.2.5 Pipette, 10 ml.

A-3.2.6 Burette, 50 ml.

A-3.3 Reagents

Unless otherwise specified, all chemicals should be of reagent grade and distilled water should be used.

A-3.3.1 Dilute Nitric Acid, 1:1 (v/v).

A-3.3.2 Sulphamic Acid.

A-3.3.3 Dilute Ammonium Hydroxide Solution, 1:1 (v/v).

A-3.3.4 Dilute Acetic Acid, 1:4 (v/v).

A-3.3.5 Potassium Iodide Solution, 10 percent (m/v).

A-3.3.6 Starch Indicator Solution — 1 percent (m/v), freshly prepared.

A-3.3.7 Ammonium Thiocyanate.

A-3.3.8 Standard 0.1 N Copper Solution — weigh accurately about 0.635 g of 99.9 percent pure copper granules in a 100 ml volumetric flask. Add 20 ml of 1:1 Nitric acid to dissolve. Boil the solution to expel the NO₂ fumes. Add a few lumps of sulphamic acid and gently boil the solution. Cool and make up to 100 ml with water.

A-3.3.9 Standard 0.1 N Sodium thiosulphate solution — weigh about 25.3 to 25.8 g of 99 percent pure Na₂S₂O₃·5H₂O in a 250 ml beaker. Add water to dissolve it. Filter into 1 000 ml volumetric flask and make up to 1 000 ml using water. Add 2-3 drops of chloroform to preserve the solution against deterioration on aging.

A-3.4 Standardization of Sodium Thiosulphate Solution Against Copper Solution

Pipette out 10 ml of Standard Copper solution (see A-3.3.8) in to a 250 ml conical flask. Add 1:1 ammonium hydroxide solution drop by drop till a faint precipitate or deep blue color appears. Then add 1:4 acetic acid drop wise until the faint precipitate or deep blue color disappears. Add 25 ml of 10 percent Potassium iodide solution. Immediately titrate the brownish solution with standard 0.1 N sodium thiosulphate solution to a pale-straw color. Add 5 ml of starch indicator solution and add 2 g of ammonium thiocyanate. Continue the titration. End point is the disappearance of blue colour. Note the volume of sodium thiosulphate solution.

$$\text{Normality of sodium thiosulphate solution} = \frac{N_1 \times V_1}{V_2}$$

where

N_1 = normality of standard copper solution,

V_1 = volume of standard copper solution, and

V_2 = volume of sodium thiosulphate solution.

A-3.5 Procedure

Weigh accurately about 0.80 g of sample in a 100 ml volumetric flask (W). Add 20 ml of 1:1 Nitric acid to dissolve. Boil the solution to expel the NO₂ fumes. Add a few lumps of sulphamic acid and gently boil the solution. Cool and make up to 100 ml with water. Pipette out 10 ml of the solution in to a 250 ml conical flask. Add 1:1 ammonium hydroxide solution drop by drop till a faint precipitate or deep blue color appears.

Then add 1:4 acetic acid drop wise until the faint precipitate or deep blue color disappears. Add 25 ml of 10 percent potassium iodide solution. Immediately titrate the brownish solution with standard 0.1 N sodium thiosulphate solution to a pale-straw color. Add 5 ml of starch indicator solution and add 2 g of ammonium thiocyanate. Continue the titration. End point is the disappearance of blue colour. Note the volume of Sodium thiosulphate solution (V).

1 ml of 1 N sodium thiosulphate solution = 0.063 546 g of copper

A-3.6 Calculation

Total copper content, percent by mass =

$$\frac{V \times N \times 0.063\ 546 \times 100 \times 100}{10 \times W}$$

where

V = volume of the standard sodium thiosulphate solution required for the test with the material, in ml;

N = normality of standard sodium thiosulphate solution,

W = weight of sample taken for the test, in g.

A-4 DETERMINATION OF CUPROUS OXIDE

A-4.1 General

Cuprous oxide is estimated by determining the Total Reducing Power (TRP) by dissolving the sample in acidulated ferric chloride solution and titrating with standard potassium dichromate solution. But occasionally, some metallic copper may be present in the sample and it gets involved in the determination of cuprous oxide. In such a case advantage is taken of the fact that cuprous oxide is soluble in cold aqueous ammonia, whereas cupric oxide is slightly soluble and metallic copper insoluble. The addition of hydrazine sulphate to the reagent (aqueous ammonia) tends to reduce any divalent copper entering in to the solution and prevents interaction of cupric oxide and metallic copper. If the extraction is carried out in carbon dioxide atmosphere, the metallic copper-cupric oxide mixture may be separated and the metal determined by solution in acidulated ferric chloride, followed by titration with potassium dichromate.

A-4.2 Determination of Metallic Copper

A-4.2.1 Apparatus

A-4.2.1.1 Stoppered-conical flask, 250 ml.

A-4.2.1.2 Glass weighing tube, closed at one end, about 35 mm long and having an internal diameter of 8 mm.

A-4.2.1.3 Glass beads

A-4.2.1.4 Whatmann filter paper No. 41, 125 mm diameter.

A-4.2.2 Reagents

Unless otherwise specified, all chemicals should be of reagent grade and distilled water should be used.

A-4.2.2.1 Ethyl alcohol, 95 percent by volume.

A-4.2.2.2 Hydrazine sulphate.

A-4.2.2.3 Liquor Ammonia — having specific gravity = 0.9.

A-4.2.2.4 Ferric chloride — $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

A-4.2.2.5 Concentrated hydrochloric acid

A-4.2.2.6 Orthophosphoric acid, 85 percent by volume.

A-4.2.2.7 Diphenyl amine indicator solution — Dissolve 0.1 g of diphenyl amine in 10 ml of freshly prepared concentrated sulphuric acid.

A-4.2.2.8 Standard 0.1 N Potassium dichromate solution — Heat fine powder of potassium dichromate in an air oven at 140-150 °C for 30 min. Cool and weigh accurately 4.9 g and dissolve it with water. Transfer in to 1 000 ml volumetric flask and make up to 1 000 ml with distilled water.

A-4.2.2.9 Air-free water, saturated with CO_2 — Boil water vigorously for several minutes and allow to cool in an atmosphere of carbon dioxide by passing a slow stream of gas continuously through the storage bottle.

A-4.2.2.10 Extraction solution — Dissolve 6 g of hydrazine sulphate in 1 l of liquor ammonia.

A-4.2.2.11 Acidulated Ferric chloride solution — Dissolve 150 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 300 ml of concentrated Hydrochloric acid in 800 ml of air-free water saturated with carbon dioxide (see A-4.2.2.9).

A-4.2.3 Procedure

Weigh accurately about 0.25 g of sample and transfer it to a 250 ml stoppered-conical flask. Displace the air in the flask with carbon dioxide gas, the carbon dioxide gas inlet being kept about 50 mm above the surface of the liquid.

Add 10 ml of ethyl alcohol and disperse well to dissolve any oil present in the sample. Add 150 ml extraction solution (see A-4.2.2.10) and shake well to dissolve the cuprous oxide content in the sample. Allow for 5 h to complete the dissolution of cuprous oxide content in the sample, but the blackish cupric oxide and reddish metallic copper portions are found left as the insoluble residue.

Filter through a No.41 Whitman filter paper and wash with 100 ml of distilled water saturated with carbon dioxide. Five to six 100 ml portions of wash water (see A-4.2.2.9) will be sufficient to remove traces of extraction solution and dissolved cuprous oxide.

Transfer the filter paper along with the residue into the conical flask. Add 15 ml of acidulated ferric chloride solution. Add a few glass beads. Stopper the conical flask and rotate gently to dissolve the cupric oxide-metallic copper residue. Allow 2 h to complete the dissolution. When the dissolution is completed, add 50 ml of distilled water, 10 ml of orthophosphoric acid and 3 drops of diphenyl amine indicator.

Titrate at once with standard 0.1 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution until a change of colour from pea-green to intense purple (V_1). Run a blank on the reagents used (V_2).

Calculate the cuprous oxide equivalent of the metallic copper using the following relationship:

1 ml of 1 N $\text{K}_2\text{Cr}_2\text{O}_7$ is equivalent to 0.071 546 g of cuprous oxide.

A-4.2.4 Calculation

Metallic copper, as cuprous oxide, percent by mass =

$$\frac{(V_1 - V_2) \times N \times 0.071546 \times 100}{W}$$

where

V_1 = volume of standard potassium dichromate solution consumed for titration of sample solution,

V_2 = volume of standard potassium dichromate solution consumed for titration of blank,

N = normality of standard potassium dichromate solution, and

W = weight of the sample taken for test.

A-4.3 Determination of Total Reducing Power (TRP)**A-4.3.1 Apparatus**

A-4.3.1.1 Stoppered-conical flask, 250 ml.

A-4.3.1.2 Glass weighing tube, closed at one end, about 35 mm long and having an internal dia of 8 mm.

A-4.3.1.3 Glass beads.

A-4.3.2 Reagents

Unless otherwise specified, all chemicals should be of reagent grade and distilled water should be used.

A-4.3.2.1 Ferric chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$.

A-4.3.2.2 Concentrated hydrochloric acid.

A-4.3.2.3 *Orthophosphoric acid*, 85 percent by volume.

A-4.3.2.4 *Potassium dichromate*

A-4.3.2.5 *Acidulated Ferric chloride* — see **A-4.2.2.11**.

A-4.3.2.6 *Diphenyl amine indicator solution* — see **A-4.2.2.7**

A-4.3.2.7 *Standard 0.1 N Potassium dichromate solution* — see **A-4.2.2.8**

A-4.3.3 Procedure

Weigh accurately about 0.25 g of sample and transfer it to a 250 ml stoppered-conical flask. Add a few glass beads. Displace the air in the flask with carbon dioxide gas, the carbon dioxide gas inlet being kept about 50 mm above the surface of the liquid. Add 15 ml of acidulated ferric chloride solution. Stopper the conical flask immediately and rotate gently until the sample is completely dissolved. Allow 2 h to complete the dissolution.

When the dissolution is completed, add 50 ml of distilled water, 10 ml of orthophosphoric acid and 3 drops of diphenyl amine indicator. Titrate at once with standard 0.1 N $K_2Cr_2O_7$ solution until a change of color from pea-green to intense purple (V_1). Run a blank on the reagents used (V_2).

Calculate the percent of cuprous oxide using the following relationship:

1 ml of 1 N $K_2Cr_2O_7$ is equivalent to 0.071 546 g of cuprous oxide.

A-4.3.4 Calculation

Total reducing power, percent by mass =

$$\frac{(V_1 - V_2) \times N \times 0.071\ 546 \times 100}{W}$$

where

V_1 = volume of standard potassium dichromate solution consumed for titration of sample solution,

V_2 = volume of standard potassium dichromate solution consumed for titration of blank,

N = normality of standard potassium dichromate solution, and

W = weight of the sample taken for test.

A-4.4 Calculation of Cuprous Oxide Content

Cuprous oxide content, percent by mass = TRP – MC

where

TRP = total reducing power (see **A-4.3.4**).

MC = metallic copper (see **A-4.2.4**).

A-5 DETERMINATION OF CUPRIC OXIDE

A-5.1 Determine total copper content in sample as per the method described under **A-3**.

A-5.2 Determine cuprous oxide content in sample as per the method described under **A-4**. Then, calculate copper content in cuprous oxide using the following formula —

$$\text{Copper content in cuprous oxide} = \frac{127.092 \times C}{143.092}$$

where

C = Cuprous oxide content, percent by mass (see **A-4.4**).

$$\text{A-5.3 Cupric oxide, percent by mass} = \frac{(X - Y) \times 79.546}{63.546}$$

where

X = total copper present in sample, and

Y = copper content of cuprous oxide present in sample.

A-6 DETERMINATION OF CHLORIDE AND SULPHATE

A-6.1 Apparatus

A-6.1.1 *Beaker*, 250 ml (graduated to indicate approximate volume)

A-6.1.2 *Volumetric Flask*, 500 ml

A-6.1.3 *Gooch Crucible*

A-6.2 Reagents

Unless otherwise specified, all chemicals should be of reagent grade and distilled water should be used.

A-6.2.1 *Concentrated Nitric Acid*

A-6.2.3 *Dilute Nitric Acid* — 1 % (v/v)

A-6.2.4 *Silver Nitrate Solution* — 5 % (m/v)

A-6.2.5 *Sodium Carbonate Solution* — saturated solution free from chloride and sulphate

A-6.2.6 *Dilute Hydrochloric Acid* — 1:1 (v/v)

A-6.2.7 *Barium Chloride Solution* — 10 % (m/v)

A-6.3 Procedure

Weigh accurately 10 g of sample in a 250 ml beaker. Add 100 ml of water and 40 ml of concentrated nitric acid. Boil to dissolve and cool. Dilute to 150 ml with water. Add an excess of sodium carbonate solution. Boil and cool.

Transfer to a 500 ml volumetric flask and make up the volume with distilled water. Allow the precipitate to settle. Draw off 2 separate 50 ml aliquots of the clear solution in beaker. Make each slightly acidic with nitric acid.

A-6.4 Determination of Chloride

A-6.4.1 To one of the 50 ml aliquot, add a few drops of silver nitrate solution. If no precipitate forms, report no chlorides present. If precipitate forms, add slowly an excess of silver nitrate solution. Heat to boiling and cool. After the precipitate has settled, filter through a tared Gooch glass crucible. Wash with 1 percent nitric acid solution. Dry at 130 °C. Cool in a desiccator and weigh.

A-6.4.2 Calculation

$$\text{Chloride, percent by mass} = \frac{35.453 \times M_1 \times 500 \times 100}{143.321 \times 50 \times M_2}$$

where

M_1 = mass of silver chloride precipitate, in g, and

M_2 = mass of sample taken for test, in g.

A-6.5 Determination of Sulphate

To one of the 50 ml aliquot, add a few drops of barium chloride solution. If no precipitate forms, report no sulphates present. If precipitate forms, add slowly an excess of barium chloride solution. Heat to boiling and cool. After the precipitate has settled, filter through a tared Gooch glass crucible. Wash with 1 percent hydrochloric acid solution. Dry at 130 °C. Cool in a desiccator and weigh.

A-6.5.1 Calculation

$$\text{Sulphate, percent by mass} = \frac{96.06 \times M_1 \times 500 \times 100}{233.39 \times 50 \times M_2}$$

where

M_1 = mass of barium sulphate, in g, and

M_2 = mass of sample taken for test, in g.

A-7 DETERMINATION OF IRON AND LEAD

A-7.1 Apparatus

A-7.1.1 Atomic Absorption Spectrophotometer — The manufacturer's instructions shall be followed for performing AAS instrumental analysis.

A-7.1.2 Beaker, 250 ml.

A-7.1.3 Volumetric flask, 100 ml and 1 000 ml.

A-7.2 Reagents

A-7.2.1 Concentrated Hydrochloric Acid

A-7.2.2 Concentrated Nitric Acid

A-7.2.3 Dilute Nitric Acid — 1:1 (v/v)

A-7.2.4 High Purity Iron Granules — at least 99 percent pure.

A-7.2.5 High Purity Lead Metal — at least 99 percent pure.

A-7.3 Preparation of Standard Stock Solutions

A-7.3.1 Standard Stock Solution of Iron

Dissolve 1 g of iron granules in 50 ml of 1:1 Nitric acid. Boil to expel nitrous fumes and cool. Make up the solution in 1 000 ml volumetric flask with distilled water. Each 1 ml of solution contains 1 000 ppm of Iron. Make appropriate dilution to prepare the standard solutions of required concentrations.

A-7.3.2 Standard Stock Solution of Lead

Dissolve 1 g of lead metal in 50 ml of 1:1 nitric acid. Boil to expel nitrous fumes and cool. Make up the solution in 1 000 ml volumetric flask with distilled water. Each 1 ml of solution contains 1 000 ppm of Lead. Make appropriate dilution to prepare the standard solutions of required concentrations.

A-7.4 Procedure

Take 1 g of accurately weighed sample in a 100 ml volumetric flask. Add 15 ml of water. Add 15 ml of concentrated nitric acid in small portions to dissolve the sample, taking sufficient precautions to avoid any vigorous reaction. Boil to dissolve and then cool. Make up to 100 ml with water.

This test solution is fed in AAS for the determination of iron and lead. Prepare an acid blank. Determine the concentration of the element of interest using atomic absorption spectrophotometer. The relevant wavelength for iron is 248.3 nm and lead is 283.3 nm.

A-7.5 Calculation

The concentration of element of interest obtained using atomic absorption spectrometer is in terms of parts per million. From this, the percent of element of interest can be calculated using the following equation —

$$\text{Concentration of the element (iron or lead), \%} = \frac{c}{100 \times m}$$

where

c = concentration of the element, in ppm.

m = mass of sample taken for test, in g.

ANNEX B

(Clause 9)

SAMPLING OF CUPRIC OXIDE

B-1 GENERAL

In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possibly by suitable means.

B-1.5 The samples shall be placed in clean, dry, airtight glass or other suitable containers which have no action on the material.

B-1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

B-1.7 Each container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* 7) and the date of sampling.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2 SCALE OF SAMPLING**B-2.1 Lot**

All containers in a single consignment of the material of the same grade and belonging to the same batch of manufacture shall constitute a lot.

B-2.2 For ascertaining the conformity of material to the requirements of the standard, samples shall be tested from each lot separately.

B-2.3 The number of containers to be selected shall depend on the size of the lot and shall be in accordance with Table 2. These containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3** draw portions of material from different parts of each container. The quantity of material so drawn shall be sufficient for triplicate determination of all characteristics given in 5.

B-3.2 Thoroughly mix portions of material drawn from each container. Out of each of these portions, approximately equal quantity of material shall be taken and mixed thoroughly to form a composite sample. The composite sample shall be divided into three equal parts; one for the purchaser, another for the supplier and the third to be used as referee sample. Each of these parts of composite sample shall be immediately transferred to thoroughly dried bottles which are sealed air-tight with stoppers. These shall be labelled with all the particulars of sample given under 7.

B-3.3 The referee sample bearing the seals of purchaser and supplier shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

B-4 NUMBER OF TESTS

All characteristics given in 5 shall be tested on the composite sample.

B-5 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of the standard if all the test results on the composite sample meet the corresponding requirements given in 5.

Table 2 Scale of Sampling

(Clause B-2.3)

Sl No.	Lot size	No. of Containers to be Selected
(1)	(2)	(3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 150	6
v)	151 to 300	7
vi)	301 to 500	8
vii)	501 to 1 000	9
viii)	1 001 and above	10

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Explosives and Pyrotechnics Sectional Committee, CHD 26

<i>Organization</i>	<i>Representative</i>
Petroleum and Explosives Safety Organization (PESO), Nagpur	SHRI M. K. JHALA (Chairman)
Arumugam Fireworks Pvt Ltd, Sivakasi	SHRI K. MARIAPPAN SHRI ARUN LALITH KUMAR (<i>Alternate</i>)
Ayyan Fireworks, Sivakasi	SHRI G. ABIRUBEN
CDET Explosives Industries Pvt Ltd, Nagpur	SHRI RAGHAV RATHI SHRI SAUMITRA RATHI (<i>Alternate</i>)
Central Institute of Mining and Fuel Research, Dhanbad	SHRI R. R. SINGH SHRI C. SAWMLIANA (<i>Alternate</i>)
Central Mine Planning and Design Institute Ltd, Ranchi	SHRI MANOJ KUMAR JHA SHRI BINAY KUMAR SINGH (<i>Alternate</i>)
Central Pollution Control Board, New Delhi	SHRI ABHIJIT PATHAK
Coal India Ltd, Kolkata	SHRI NRIPENDRA NATH
Consumer Guidance Society of India	SHRI SITARAM DIXIT DR M. S. KAMATH (<i>Alternate</i>)
Defence Standardization Cell (DRDO), Ministry of Defence, New Delhi	DR H. L. YADAV MS HEMLATA GAUTAM (<i>Alternate</i>)
Departmental Testing Station, PESO, Nagpur	SHRI R. P. PIPLANI
Directorate General of Mines Safety, Dhanbad	SHRI C. RAMESH KUMAR SHRI S. S. SONI (<i>Alternate</i>)
Directorate of Standardization, New Delhi	SECRETARY, ASSC/JD (STD)
ENif Paper Products Pvt Ltd	SHRI MANAN DESAI
Fireworks Manufacturers Association (North India), Gwalior	SHRI HARISH MILWANI SHRI RAJEEV JAIN (<i>Alternate</i>)
Fireworks Research & Development Centre, Sivakasi	SHRI AMIT GOEL
Hariwansh Packaging Pvt Ltd	SHRI VIJAY MURARKA
Gudiya Fireworks, Delhi	NOMINATION AWAITED
GOCL Corporation Ltd, Hyderabad	SHRI C. SAINATH SHRI P. SITARAMASWAMY (<i>Alternate</i>)
High Energy Materials Research Laboratory, Pune	SHRI C. GURURAJA RAO SHRI M. P. KULKARNI (<i>Alternate</i>)
Ideal Wrappers, Nagpur	SHRI KISHORE JUNHERS
IDL Explosives Ltd., Hyderabad	SHRI N. VIDYASAGAR SHRI A. K. DASGUPTA (<i>Alternate</i>)
Ministry of Defence (DGQA), New Delhi	SHRI R. K. RAMTEKE DR R. N. APARAJIT (<i>Alternate</i>)
Ministry of Defence, Ordnance Factory, Bhandara	DR ONKAR MONDHE D. K. URKUDE (<i>Alternate</i>)
Neyveli Lignite Corporation Ltd.	SHRI M. MUTHUKUMARAN

<i>Organization</i>	<i>Representative</i>
Petroleum and Explosives Safety Organization (PESO), Nagpur	SHRI R. A. GUJAR
Shri Kaliswari Fireworks, Sivakasi	SHRI A. P. SELVARAJAN
Solar Industries India Ltd, Nagpur	SHRI R. K. GUHA SHRI A. K. JAIN (<i>Alternate</i>)
Standard Fireworks, Sivakasi	SHRI M. S. SARAVANAN
The All India Chamber of Match Industries, Sivakasi	THE PRESIDENT THE SECRETARY (<i>Alternate</i>)
The Coronation Fireworks Factory, Sivakasi	SHRI P. KANAGAVEL SHRI K. JEYAKUMAR (<i>Alternate</i>)
The Indian Fireworks Manufacturers Association, Sivakasi	SHRI T. KANNAN THE SECRETARY (<i>Alternate</i>)
The Metal Powder Co Ltd, Thirumangalam	SHRI P. SUNDARAPANDIAN SHRI N. CHANDRASEKARAN (<i>Alternate</i>)
The Singareni Collieries Co Ltd, Kothagudem	SHRI S. N. SIVA KUMAR
The Tamil Nadu Fireworks and Amorce Manufacturers Association, Sivakasi	THE PRESIDENT THE SECRETARY (<i>Alternate</i>)
WETWO Fireworks, Sivakasi	SHRI G. V. SHYAMSUNDAR
Yogeshwar Packers, Nagpur	SHRI Y. K. YADAV
BIS Directorate General	SHRI U. K. DAS, SCIENTIST 'E' AND HEAD (CHD) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary

SHRI SACHIN S. MENON
SCIENTIST 'B' CHD, BIS

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This Indian Standard has been developed from Doc No.: CHD 26 (11924).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi